Supplementary Materials for

Isotope Ratios of H, C, and O in CO_2 and H_2O of the Martian Atmosphere

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Materials and Methods

Spectral Data Processing

Spectral scan regions for TLS are shown in Fig. 1 and line parameters from HITRAN *(36)* are given in Tables S1 and S2 below. After normalization to the laser power and zero light pulse, spectral lines are processed individually by integrating over the line shape; line ratios are then related to those expected from calibration runs, using the HITRAN parameters if necessary to adjust for minor temperature differences. Details follow.

Theory

The Beer-Lambert law models the optical transmission of light through an absorbing medium:

$$I_\nu = I_0 e^{-k(\nu)\rho l}$$

where I_ν is the transmitted light intensity at frequency ν, I_0 is the incident light intensity, $k(\nu)$ is a line shaping function that may be Doppler, Lorenzian, or Voigt, although the Doppler lineshape is a close approximation at Mars atmospheric pressures. ρ is the number density and l is the path length in cm. We use this model to determine the abundances of individual absorption lines present in our sampled measurements and subsequently use those abundances to produce isotopic ratios. The model needs many input spectral parameters for temperature dependence, air broadening, ground state energy, etc., and we use the HITRAN database for this information *(36)*.

Normalization

For an amount of gas at a given pressure and temperature, the model will predict the depth and width (distribution in wave number) of the absorbing molecules in the gas sample for all sampled frequencies, allowing us to then compare our recorded spectra to the spectra produced by the model. But, in order to make this comparison, we must first normalize the recorded data. This entails:

1. Removing a "null pulse" which is a measurement of the background light taken with the laser off. We must be able to determine the direct absorption with respect to a percentage of transmitted light (i.e. 1% absorption: 99% transmission).
2. Removing any DC offsets in the harmonic spectra (described below).
3. Fit the baseline of the spectra. This sloping baseline results from the fact that the laser output power increases as it tunes through different wave numbers.
4. Assign a wave number (cm^{-1}) scale to the real spectra. We do this by using easily identifiable peaks of known wave number.

Once the spectra are normalized, we can then use the model to scale our real world data. Direct absorption spectra produce good results for gases that have line center absorption depths of 0.5% or greater. For greater resolution, we add a modulation to the laser current and then demodulate the returning detector signal at twice that frequency. This effectively gives us a second derivative of the direct spectra. Using this derivative or *2f* method, sensitivities of up to 2 parts in 10^5 are possible. See Webster et al. for a complete discussion *(37)*.

Producing Abundances

Using temperatures and pressures from our instrument for input, we iteratively run the model, varying the abundance in a converging algorithm until the synthetic spectra is the same size as our real spectra (within some determined threshold). The convergence criteria may be set to optimize for either the direct absorption or 2f spectra.

The algorithm is as follows (see Figs. S1 and S2):

1. Find the global max of the 2f absorption spectra (peak)
2. Find the two local minima (2f lobes)
3. Fit a line between the two lobes
4. Using the lobes as integration boundaries, find the area between the fitted line and the spectra for both the direct and 2f spectra. Ratio this area between real and synthetic spectra and if ratio is outside the convergence threshold, iterate with new abundance.

Once the measurements converge, we ratio the resulting areas of the real spectra to the synthetic spectra which has a known abundance. We do this for both the direct and harmonic spectra. In general, the direct spectra provide us with accuracy, since it is a very simple percentage measurement. The harmonic spectra, with its great sensitivity, provide greater precision for small changes in the signal, although determining the exact modulation values and gains introduce opportunity for error.

Although it is common practice to fit the entire spectra simultaneously, certain artifacts in our spectra make that approach problematic (see Instrument Issues section below). Instead, we process individual absorptions separately, which is not difficult at these Doppler-limited low pressures where the absorption lines tend to be clearly delineated. The benefits of this approach are 1) that we use the signal where it is strongest (between the 2f lobes), 2) it weakens the dependency on perfect baseline fitting, and 3) the boundaries of integration are set by the physics of absorption, rather than relying on a perfect wave number assignment.

Producing Isotope Ratios

Once we repeat this for every line of interest, we can create isotopic ratios for each data point (2 minute spectrum) according to the following standard isotope ratio formula for returning enrichment or depletion ratios in per mil. As an example, consider the case for comparing abundances of ^{13}C to ^{12}C in CO_2 to calculate $\delta^{13}C$ in units of per mil:

$$\left(\left[\frac{{}^{13}CO2\_Observed \,/\, {}^{12}CO2\_Observed}{{}^{13}CO2\_Standard \,/\, {}^{12}CO2\_Standard}\right] - 1\right) * 1000$$

Instrument Issues

The HITRAN database reports its parameters to a few percent and we have refined some of those parameters through calibration testing. As for the artifacts mentioned above, several signal chain filters were set to values that attenuated the signal according to frequency, producing a “ring” on the direct absorption line shape. Fig. 2 in the main paper illustrates this effect, where part of the spectral region 1 is shown for CO_2 lines and is compared with the HITRAN calculations. Rather than try to remove the effects of that filter in our data, we build a software filter with identical behavior and make fine adjustments until our modeled output matches the line shape ring of our Mars spectra. In this way, we compare our Mars spectra with HITRAN-generated spectra that now have line shape rings included. Another potential issue is related to the susceptibility of the NIR laser to wavelength drift with heat sink temperature that depends on the Mars conditions (time of day). Across most of the scan range the laser tuning is constant and the effect is cancelled out. But we are watchful for situations where lines of interest are in the beginning of the scan where laser tuning rate is changing, and in these situations data is either discarded or the individual line results are given appropriately higher uncertainties.

Calibration

Calibration of the relative absorptions of isotopic pair lines was done pre-launch using either commercially-provided certified isotopic gas mixtures (Oztech) or specially prepared tanks (Cylinders A, B) whose isotopic content was determined by Isotope Ratio Mass Spectrometry (IRMS). For water isotopes, we used "Boulder water" independently certified by NOAA. See Table S3 for calibration gas isotopic values. Minor temperature and pressure interpolations are done using the HITRAN *(36)* line list. Although spectral SNR's are typically a few thousand, the data show scatter larger than this, and the reported results for any single run (Sol) are a mean value and either 1- or 2-standard errors from the mean (SEM) on the results (see Tables 1 and S4). Figure S3 shows our calibration plot of pre-launch and on-Mars cal gas results vs. retrieved measurements to show the excellent linearity over the measurement region.

Supplementary Text

Isotope Ratio Results

Tables of results on a sol-by-sol basis are given in Table S4. Because there is little water in the Martian atmosphere, the results for the δD values have quite high uncertainties compared to the result for the water evolved at lower temperatures from the Rocknest fines *(23)*.

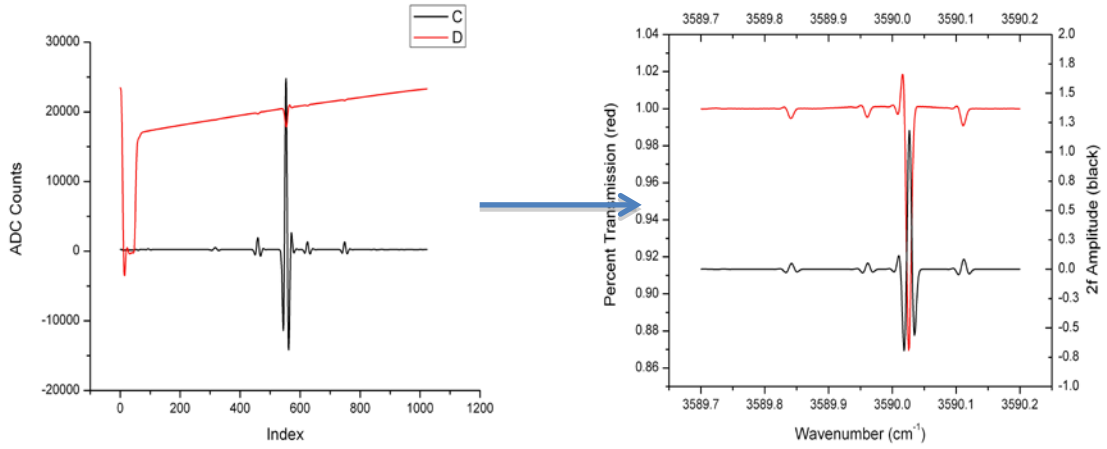

Fig. S1.
Example of normalization of real spectra for region 1. Lines can be identified from Fig. 1 in the main paper. The "C" or black traces are the second harmonic (2f) spectra, while the "D" or red traces are the direct absorption (DC) spectra.

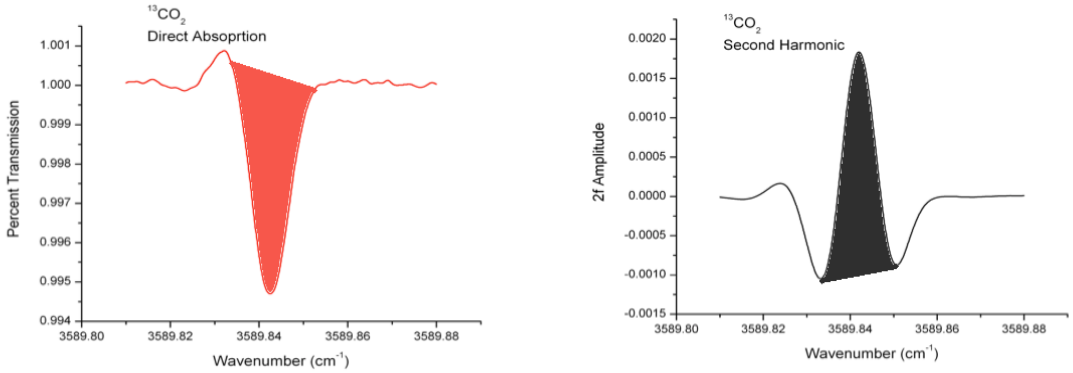

Fig. S2

Examples of direct (left) and 2f (right) line shapes for a single $^{13}CO_2$ line, showing the integrated area used in retrievals. This area is defined by the location of the two minima (lobes) in the 2f line shape mapped to the wave number scale in both line shapes.

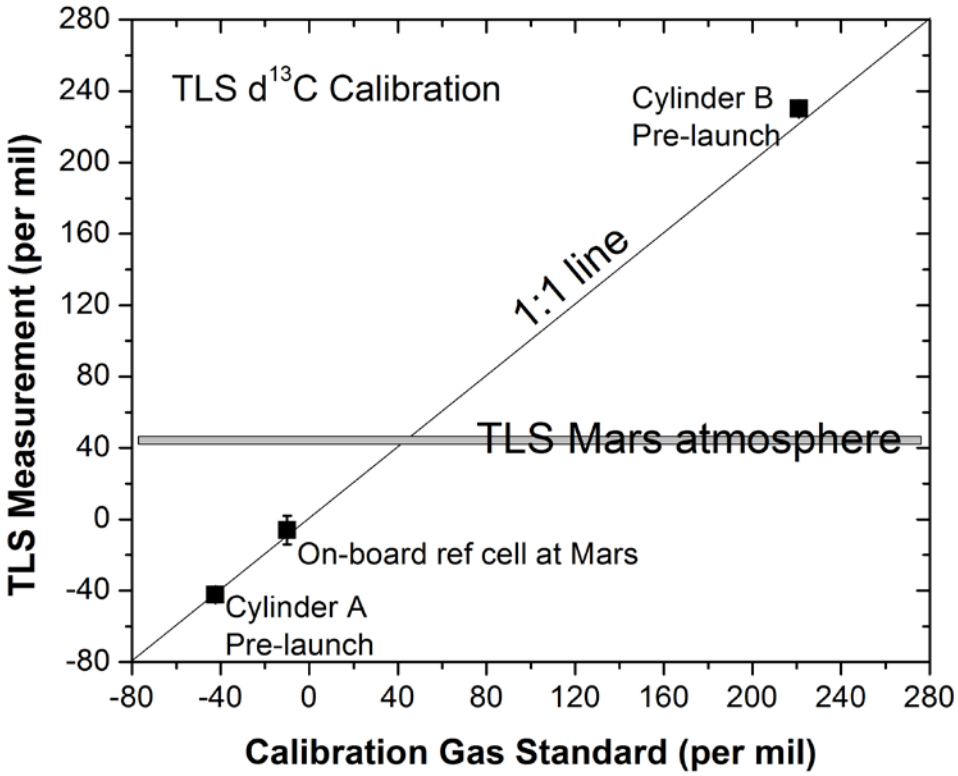

Fig. S3

Calibration plot showing pre-launch and on-Mars cal gas results vs. retrieved measurements to show excellent linearity over the measurement region.

Table S1.

HITRAN Line Parameters *(36)* for Spectral Region 1.

Line Designation	**Isotopic Species**	**Wavenumber (cm^{-1})**	**Linestrength (cm^{-1}/ (molecule·cm^{-2}) at 296 K)**	**Ground State Energy (cm^{-1})**
Z (H2O)	H_2O	3590.238310	1.222E-21	206.3014
X (H2O)	H_2O	3590.165390	5.164E-24	2251.8625
A (CO2)	CO_2	3590.116079	5.734E-23	1648.4209
Y (H2O)	HDO	3590.072290	4.478E-25	683.3240
B (CO2)	CO_2	3590.031510	1.341E-21	728.4124
C (CO2)	^{18}OCO	3589.966048	6.399E-23	319.8096
D (CO2)	$^{13}CO_2$	3589.846500	5.180E-23	843.0702
E (CO2)	^{17}OCO	3589.749440	3.057E-24	2.2717
F (H2O)	H_2O	3589.590960	4.599E-23	1437.9686

Table S2.

HITRAN Line Parameters *(36)* for Spectral Region 2.

Line Designation	**Isotopic Species**	**Wavenumber (cm^{-1})**	**Linestrength (cm^{-1}/ (molecule·cm^{-2}) at 296 K)**	**Ground State Energy (cm^{-1})**
Z (CO2)	^{18}OCO	3594.279835	3.294E-23	490.2111
Y (CO2)	CO_2	3594.216942	1.392E-21	801.1732
X (CO2)	$^{13}CO_2$	3594.160247	9.647E-23	704.3340
B (H2O)	$H_2{}^{18}O$	3594.061450	1.827E-23	78.9887
C (H2O)	H_2O	3593.974520	4.962E-22	756.7248
S (CO2)	$^{18}O^{13}CO$	3593.907244	9.883E-25	26.5089
W (CO2)	CO_2	3593.819208	3.554E-23	1805.2522
D (H2O)	H_2O	3593.791190	5.325E-24	382.5169
V (CO2)	^{18}OCO	3593.762005	3.640E-23	463.7240
E (H2O)	HDO	3593.597530	4.881E-24	513.2072
F (H2O)	$H_2{}^{18}O$	3593.545310	6.132E-24	133.4758
U (CO2)	^{17}OCO	3593.489040	4.590E-24	2.2717
T (CO2)	CO_2	3593.406043	1.411E-21	786.9028
G (H2O)	HDO	3593.317210	4.922E-24	512.5158
H (H2O)	H_2O	3593.197361	1.719E-02	602.7735

Table S3.

Calibration Standards Used in TLS as determined by Isotope Ratio Mass Spectrometry (IRMS).

Carbon Dioxide Isotope Values:		
Standard	$\delta^{13}C$	$\delta^{18}O$
Cylinder A	-43.02 ± 0.01	6.62 ± 0.01
Cylinder B	220.91 ± 0.05	476.05 ± 1.72
Cylinder C	475.64 ± 0.24	794.23 ± 9.9
Mars mix	-39.16 ± 0.05	0.61 ± 0.07
Oztech (ref cells)	-10.43 ± 0.05	31.24 ± 0.05
Water Isotope values:		
Standard	δD	$\delta^{18}O$
Boulder water	-110.11± 0.05	-14.91± 0.05

Table S4.

Sol by sol mean isotope ratio values (‰) and uncertainties (1_{SEM} ‰) measured by TLS. The first row for each entry is from Region 1, and the second row from Region 2. An "X" marks no reliable data retrieved due to SNR or other instrument issues. PTC = Possible Terrestrial Contamination; PC = Post-pyrolysis contamination.

Sol	**$\delta^{13}C\_CO_2$**	**$\delta^{18}O\_CO_2$**	**$\delta^{17}O\_CO_2$**	**$\delta^{13}C^{18}O\_CO_2$**	**δD_H_2O**
28	53.1 ± 5.7 X	41.5 ± 9.2 53.3 ± 2.8	**X** 22.7 ± 6.0	X 147 ± 12	PTC
53	45.3 ± 3.7 39.03 ± 5.7	43.4 ± 5.3 36.48 ± 6.3	30.6 ± 3.6 8.4 ± 7.6	X X	PTC
73	34.3 ± 3.4 49.85 ± 4.0	39.9 ± 7.5 38.3 ± 7.3	28.5 ± 5.2 X	X 114 ± 27	4,420 ± 430
79	41.1 ± 4.4 50.8 ± 11	50.5 ± 4.0 63.2 ± 9.6	32.1 ± 5.3 17.7 ± 15	X 112 ± 52	5,480 ± 980
106	47.1 ± 1.9 50.1 ± 5.0	61.6 ± 3.7 47.3 ± 4.0	18.0 ± 2.9 10.2 ± 6.0	X 62 ± 18	PC
Range of values	34.3-53.1	36.5-63.2	8.4-32.1	62-147	4,420-5,480
Mean $\pm1_{SEM}$	45.8 ± 2.1	47.6 ± 2.4	24.2 ± 2.0	109 ± 15.6	4,950 ± 540
Mean $\pm2_{SEM}$	**46 ± 4**	**48 ± 5**	**24 ± 5**	**109 ± 31**	**4,950 ± 1,080**

Reference:

37. C. R. Webster, R. T. Menzies, and E. D. Hinkley, in *Laser Remote Chemical Analysis*, R. M. Measures, Ed. (Wiley, New York, New York) chap. 3, (1988).

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